

add info for Log 440

L. Lister
WRW

slh
C-2135



Amoco Oil Company
200 East Randolph Drive
Post Office Box 6110-A
Chicago, Illinois 60680

October 7, 1983

Mr. Myles E. Morse
Environmental Protection Specialist
Waste Characterization Branch (WH-565 B)
U. S. Environmental Protection Agency
401 M Street, SW
Washington, D. C. 20460

Dear Sir:

Additional Information and Data - Amoco
Petition for Exclusion from Regulation

Enclosed is a complete response to your August 11, 1983 request for additional analytical data and information pertaining to our plans for chemically stabilizing (Chemfixing) DAF float (K048) presently stored in surge ponds at the site of our former refinery in Wood River, Illinois. These data are in support of our petition to exclude the Chemfixed product from regulation. We are resubmitting the analytical data that weremailed to you as a partial response on October 4, 1983.

The enclosures include the following:

- Enclosure I - Organics contents and other test data on two Chemfixed quarter-pond composites from Wood River surge ponds. Includes total organics and organics leachable by the EP toxicity test. Includes data on surrogate spiking. (Item 3).
- Enclosure IIA - Copy of our February 28, 1983 letter discussing sample representativeness. (Item 1).
- Enclosure IIB - Copy of our August 9, 1983 letter presenting data on fifteen additional grab samples from the surge ponds. (Item 1).
- Enclosure III - Response to questions regarding method of standard additions, the Chemfix process, and the long term stability of Chemfixed material. (Items 2, 4, and 5).

Except for the analytical data, the responses are consistent with the requests in your letter and require no explanation.

RECEIVED

OCT 11 1983

EPA - D.C.P.C.
STATE OF ILLINOIS

EPA Region 5 Records Ctr.



291060

Mr. Myles E. Morse
10/7/83

- 2 -

Variations between analytical data supplied in the enclosed tables and data requested in your letter resulted from numerous telephone conversations between Agency and Amoco personnel. In conversations with you and Mr. Jim Poppiti, our Dr. R. F. Babcock was requested to add tests for total phenols, total cresols, and tetraethyl lead and to add hydroquinone, naphthoquinone, quinoline, and isoquinoline to the list of organics for analysis by gas chromatography/mass spectroscopy (GC/MS). In our earlier conversation with you, I was instructed to remove "process specific compounds" from the list of volatile organics for GC/MS analysis.

Extraction procedures for the samples and the compounds for surrogate spiking were discussed and agreed upon by Mr. Poppiti and Dr. Babcock. Analytical methods used were those in "Test Methods for Evaluating Solid Waste" (SW-846). Unusually high detection limits in some cases of GC/MS analysis resulted from a particular combination of sample size and final solution volume. These limits will be lowered for any future work.

We are looking forward to discussing the enclosed information in our meeting on October 12, 1983.

Yours truly,



E. J. Sullivan
Environmental Consultant
Mail Code 1203

EJS:ab

Enclosures

cc: Mr. Larry Eastep - IEPA

RECEIVED

OCT 11 1983

IEPA - DLR/C.
STATE OF ILLINOIS

TABLE VI

Methods Used for Wood River Sludge Analysis

Total Volatile Organics--0.40 gm of sample was mixed with 5 ml of polyethylene glycol in a 40-ml vial, which was then filled with deionized water and capped with no head space. 5 ml of the sample from the vial was analyzed by purge and trap GC/MS.

Leachable Volatile Organics--A VOA vial was filled with filtered EP toxicity leachate. 5 ml of the vial sample was analyzed by purge and trap GC/MS.

Total Nonvolatile Organics--5 gm of sample was Soxhlet extracted for 4 hours with 150 ml of methylene chloride. The extract was concentrated to 5 ml and analyzed by capillary column GC/MS.

Leachable Nonvolatile Organics--500 ml of EP toxicity leachate was extracted with methylene chloride for base/neutral and acidic organics. The extracts were concentrated to 1 ml and analyzed by capillary column GC/MS.

Total Phenols and Cresols--10 gm of sample was mixed with 100 ml of deionized water and distilled at pH 4. A portion of the distillate was analyzed for total phenols by the 4-amino-antipyrene colorimetric method. Another portion of the distillate was extracted at pH 4.0 with hexane to remove cresols, and the aqueous fraction was analyzed as above for phenols. Cresols were determined by the difference between the total phenols before and after hexane extraction at pH 4.0.

TEL--10 gm of sample was mixed with 100 ml of 1 + 1 toluene-xylene and distilled. The fraction boiling above 133°C was analyzed for lead by ICP (ASTM D-1949).

TOC--Total carbon determined by combustion, carbonate determined by titration. Organic carbon determined by difference.

Oil and Grease--Soxhlet extraction using freon as the extractant.

TABLE III

**Total Organics in Chemifixed Samples
From Wood River Surge Ponds**

Total Volatile Organics:

<u>Compound</u>	<u>Concentration, PPM</u>	
	<u>Pond 1, Quadrant 1</u>	<u>Pond 2, Quadrant 1</u>
Benzene	0.3	0.4
Chloroform	0.2	0.3
Ethylbenzene	1.5	2.3
Methylene Chloride	2.3	1.2
Toluene	1.4	1.4
M-Xylene	6.6	10.4
O-Xylene + P-Xylene	6.3	10.5

Note: No other Table-II volatiles were detected at a detection limit of 0.2 ppm.

Total Nonvolatile Organics:

<u>Compound</u>	<u>Concentration, PPM</u>	
	<u>Pond 1, Quadrant 1</u>	<u>Pond 2, Quadrant 1</u>
Acenaphthene	3	5
Acenaphthylene	ND @ 2 ppm	2
Benz (A) Anthracene	12	ND @ 2 ppm
Benz (A) Pyrene	3	ND @ 2 ppm
Benzo (K) Fluoranthene	7	2
2-Chloronaphthalene	3	ND @ 2 ppm
Chrysene	5	7
Di-N-Butylphthalate	2	2
Fluorene	8	ND @ 2 ppm
Naphthalene	18	40
Phenanthrene	31	44
Pyrene	15	12
2,4-Dichlorophenol	7	ND @ 7 ppm
Phenol	3	ND @ 5 ppm

Note: No other Table-II nonvolatiles were detected at the following detection limits:

Base-Neutrals	2 ppm
Phenol	3 ppm
Other Phenols	5 ppm

ND = Not detected at the indicated detection limit.

TABLE IV

**Organics in USEPA Extraction Procedure Leachate
From Chemfixed Samples From Wood River Surge Ponds**

Leachable Volatile Organics:

<u>Compound</u>	<u>Concentration in Leachate, $\mu\text{g}/\text{l}$</u>	
	<u>Pond 1, Quadrant 1</u>	<u>Pond 2, Quadrant 1</u>
Benzene	ND @ 10 $\mu\text{g}/\text{l}$	14
Ethylbenzene	10	11
Methylene Chloride	22	177*
Toluene	16	18
M-Xylene	43	51
O-Xylene + P-Xylene	47	55

Note: No other Table-II volatiles were detected in the leachates at a detection limit of 10 $\mu\text{g}/\text{l}$.

Leachable Nonvolatile Organics:

<u>Compound</u>	<u>Concentration in Leachate, $\mu\text{g}/\text{l}$</u>	
	<u>Pond 1, Quadrant 1</u>	<u>Pond 2, Quadrant 1</u>
Phenol	78	55
O-Cresol	36	73
Diethylphthalate	10	ND @ 10 $\mu\text{g}/\text{l}$
Naphthalene	36	34

Note: No other Table-II nonvolatiles were detected in the leachates at detection limits of 10 $\mu\text{g}/\text{l}$ for base neutral compounds and 25 $\mu\text{g}/\text{l}$ for acidic compounds.

*High concentration suspected to be the result of laboratory contamination.

RECEIVED
JAN 11 1978
STATE OF ILLINOIS

TABLE V

Results of Surrogate Spiking

Total Volatile Organics:

Pond 1, Quadrant 1:

<u>Surrogate</u>	<u>Spike Conc., ppm</u>	<u>% Recovery</u>
Fluorobenzene	5	98
3-Bromobenzotrifluoride	5	87

Total Nonvolatile Organics:

Pond 1, Quadrant 1:

<u>Surrogate</u>	<u>Spike Conc., ppm</u>	<u>% Recovery</u>
Pentafluorophenol	80	0 (1)
2-Fluorobiphenyl	80	82

- (1) Total loss of pentafluorophenol possibly due to decomposition during Soxhlet extraction, extract concentration, or chromatography. Priority pollutant phenols, phenol, and 2,4-dichlorophenol were detected in this sample.

Enclosure 117
RECEIVED

**RESPONSES TO QUESTIONS CONCERNING CHEMFIX®
PRODUCT RESULTING FROM TREATMENT
OF REFINERY WASTE SW#1227-H82
AMOCO OIL COMPANY - WOOD RIVER, IL**

OCT 11 1993

E.P.A. - D.L.R.C.
STATE OF ILLINOIS

Question 2 - At least one sample tested by the EP for chromium and lead should be analyzed using method of standard additions as a QA/QC procedure. Spikes and recoveries should be reported.

Analysis of EP Extracts for Chromium and Lead Using the Method of Standard Addition

As reported by CTI in earlier support presentations, analyses of chromium (Cr) and lead (Pb) were performed using the Method of Standard Addition specified by the EPA in Test Methods for Evaluating Solid Waste, SW-846, 2nd edition. Examples of those analyses are found in Attachment A. For each constituent-of-concern, equal volumes of EP extract were added to a deionized distilled water blank and to three standards containing different known amounts of the constituent. For both chromium and lead, the three standards used were 0.5mg/L, 1.0mg/L, and 1.5mg/L. The following EP extract concentrations were determined for the examples found in Attachment A:

<u>Product Extracted</u>	<u>Cr (mg/L)</u>	<u>Pb (mg/L)</u>
Pond 2, Quads 1 and 3	0.3	0.5
Pond 2, Quads 2 and 4	0.1	0.5

Question 4 - Submit more detailed process information on the Chemfix treatment process, identifying additives and mixing ratios.

The CHEMFIX® Process and Reagents

The patented CHEMFIX® process utilizes soluble silicates as reactants which combine with toxic metal constituents in a waste to produce insoluble metal silicates. A cementitious matrix is provided by setting agents such as Type I Portland cement. A non-hazardous solid, soil-like material is produced when reagent levels are employed in the range of 1.00 to 1.25 pounds of setting agents per gallon of waste and 2% to 4% by volume of soluble silicates per gallon of waste.

During treatment of the refinery waste, Type I Portland cement is first added to the waste and a slurry produced. Water present in the waste begins at that point to react with various calcium silicates, tricalcium aluminate, and tetracalcium aluminoferrite present in the cement to form calcium silicate hydrates, calcium aluminosilicate hydrates, and calcium hydroxide. As the silicate species are forming, ion exchange reactions begin, during which metal ions replace calcium ions in the silicate structure.

Several minutes into processing, a basic solution of sodium metasilicate is introduced into the slurry. The predominant silicate species present in the solution are polysilicate anionic branched rings. Cationic species present in the slurry begin to react with the complex silicate structures at this point. Initially, free metal ions bind covalently with polysilicate anions, followed by complex metal species binding covalently or ionically with available silicate structures. While these reactions are continuing, the metal silicate crystals continue forming larger interlocking lattice structures. Finally, non-metallic cations such as calcium and potassium form ionic bonds wherever an excess of negative charge remains.

Question 5 - Discuss the effect of long-term biodegradation of organic content and its effect on the Chemfix matrix and the metals bound in it.

Long-Term Biodegradation of Organic Content and its Effect on CHEMFIX® Product Matrix and Metals Retention

The long-term stability of CHEMFIX® product under conditions simulating 1,000 years of an acid-rain environment was determined by subjecting the products to the EPA-specified Multiple Extraction Procedure (MEP). The constituents-of-concern concentrations in each MEP extract were significantly less than maximum allowable levels and continued to decrease with subsequent extractions. These results (Attachment B) indicate the CHEMFIX® product maintains its chemical and physical stability under severe environmental conditions.

CHEMFIX® products were also subjected to the EPA-specified Extraction Procedure for Oily Waste, designed to determine whether metals will be mobilized by removal of organics. During the procedure, the products were extracted in refluxing tetrahydrofuran and toluene. The non-aqueous extracts were combined prior to analysis and the extract residues subjected to an EP. All non-aqueous as well as aqueous extracts contained significantly less than maximum allowable levels as seen below:

<u>Sample</u>	<u>Cr Concentrations (mg/l)</u>				<u>Pb Concentrations (mg/l)</u>			
	<u>Pond 1</u>	<u>Pond 2</u>	<u>Pond 3</u>	<u>Pond 4</u>	<u>Pond 1</u>	<u>Pond 2</u>	<u>Pond 3</u>	<u>Pond 4</u>
Non-Aqueous Extract	0.1	0.0	0.1	0.1	1.0	0.8	0.9	0.9
EP Extract	0.3	0.2	0.1	0.2	0.2	0.2	0.2	0.2

CHEMFIX® product long-term stability was verified by examining a refinery waste CHEMFIX® processed during a field project in 1975 which was allowed to weather in a northern environment, undergoing numerous freeze/thaw cycles. As seen below, chromium and lead were retained in the CHEMFIX® product matrix and did not leach out of the product in significant concentrations when subjected to the MEP.

1982 PROPERTIES OF A REFINERY WASTE PROCESSED IN 1975

<u>Constituent</u>	<u>1982 Digestion of CHEMFIX® Product (mg/kg)</u>	<u>1982 Extractions (mg/l)</u>									
		<u>EP</u>	<u>MEP Day1</u>	<u>MEP Day2</u>	<u>MEP Day3</u>	<u>MEP Day4</u>	<u>MEP Day5</u>	<u>MEP Day6</u>	<u>MEP Day7</u>	<u>MEP Day8</u>	<u>MEP Day9</u>
Pb	73.5	0.32	0.06	nd	nd	nd	nd	nd	nd	nd	nd
Cr	800	0.07	nd	nd	nd	nd	nd	nd	nd	nd	nd

nd = none detected

These results further illustrate that the CHEMFIX® product is non-hazardous and that metals are not in a mobile form following organic degradation or severe weathering.

APPROVED
DATE: 1/10/82
EPA - OLRD
10/10/82



H A McCandless
Vice President, Manufacturing

Madison County
Amoco
~~11911501~~ 11911502
Amoco Petroleum Additives Company
Post Office Box 182
Wood River, Illinois 62095
618-251-2200

December 21, 1983

Mr. Kenneth G. Mensing
Southern Region Manager
Land Field Operations Section
Division of Land Pollution Control
Illinois Environmental Protection Agency
117 West Main Street
Collinsville, Illinois 62234

RECEIVED
DEC 26 1983
ILL. EPA - D.L.P.C.
STATE OF ILLINOIS

Dear Sir:

Inspection of: 11911501 - Madison County - Wood River/Amoco
ILD 980700967 - Amoco (Main Plant)
ILD 980523106 - Amoco (Riverfront)
ILD 006277629 - Amoco (Waste Water Treating Plant)

Please refer to your December 5, 1983 letter concerning an inspection of the three Amoco sites made on September 22, 1983. Your letter requested a response within 15 days of receipt.

We would like to preface our remarks with a brief description of occurrences in early 1981. The situation at the Wood River plant was somewhat confusing in that both Amoco Oil and Amoco Chemicals processing units were operating on the same site. The decision to shut down Amoco Oil's refining operations was announced in February, 1981 and did not affect Amoco Chemicals operations. Thus, a part of the integrated plant stopped operating while another part continued to operate and to require ancillary services such as utilities, waste-water treating and waste disposal.

Refining operations gradually shut down and by June 1, 1981, all petroleum processing had stopped and a general cleanup had begun in preparation for "mothballing" the refinery units. Standard cleanup procedures were used and included removing wastes from certain equipment, disposing of any hazardous waste so removed in an off-site hazardous waste facility, rinsing the equipment and inspecting to confirm the equipment to be in satisfactory condition for mothballing. Closure of facilities mothballed in this fashion will be limited to certification.

RECEIVED

DEC 28 1983

ILL. EPA - D.L.P.C.
STATE OF ILLINOIS